Analysis of Lysergic Acid Diethylamide (LSD)

1. Background

Lysergic acid diethylamide (LSD), commonly referred to as "acid", is a synthetic hallucinogen. LSD is manufactured from lysergic acid, which is found in ergot, a fungus that grows on rye and other grains. It is a colorless, odorless, and tasteless liquid. It comes in a variety of forms, but is always taken orally. LSD is most commonly found of small squares of paper called blotter (full sheet of paper are decorated with artwork or designs, perforated, then soaked in liquid solution and dried). Other forms include, tablets (microdots), gelatin squares (window panes), liquid, liquid sugar cubes and powder. Additionally, LSD has been embedded in candy such as "Gummy Worms," "Sweet Tarts," "Smarties," and "Pez."

2. Objective

The objective of this SOP is to establish guidelines to be used for the analysis of a sample that may contain lysergic acid diethylamide (LSD).

3. Scope

This SOP is to be used by the laboratory staff of the Division of Analytical Chemistry at William A. Hinton State Laboratory Institute in Boston, MA.

4. Responsibility

Chemists are responsible for acquiring glassware, preparing chemical reagents and standards, sample analysis, and reporting. Chemists also perform instrument calibrations, maintenance and troubleshooting, ordering of supplies and other necessary tasks related to this analysis.

Laboratory Supervisors ensure that chemists are following this SOP. They may perform the duties of the chemists and must review raw data and reports generated by chemists. The Supervisor may advise the chemists of alternative testing methods. They ensure that quality control measures are within acceptable limits and determine when corrective actions are needed. They coordinate proficiency testing (PT), reporting and distribution of PT results. They oversee sample results distribution to outside agencies.

Directors ensure that the SOP is being followed and reviewed on a regular basis. They provide approval of standard operating procedures and review quality control documentations.

1. Related Documents

Cole, Michael, "The Analysis of Controlled Substances," London: John Wiley & Sons Ltd., 2003
Drug Enforcement Administration, "Basic Training Program for Forensic Drug Chemists," Drug Enforcement Administration.
Mills III, Terry et al, "Instrumental Data for Drug Analysis," 3rd ed., 6 vols., New York: CRC Press, 2006.
Moffat, A.C. et al, "Clarke's Isolation and Identification of Drugs," 2nd ed., London: The Pharmaceutical Press, 1986.
Moffat, A.C. et al. "Clarke's Analysis of Drugs and Poisons," 3rd ed., London: The Pharmaceutical Press, 2004.
Saferstein, Richard, "Forensic Science Handbook," New Jersey: Prentice Hall, 1988.
Scientific Working Group for the Analysis of Seized Drug Recommendation, 6th ed., "Part III A & B, Methods of Analysis/Sampling of Seized Drug for Qualitative Analysis," July 2011

6. Definitions

GC w/ FID: Gas Chromatography with Flame Ionization Detector
GC/MS: Gas Chromatography/Mass Spectrometry
Gross Weight: The weight of both the substance and its container.

7. Supplies, Equipment & Reagents

Supplies

GC columns
- HP-1MS (Agilent, Cat # 19091S-933UI or equivalent)
- HP-5MS (Agilent, Cat # 19091S-433UI or equivalent)

GC crimp vials
- Clear (Agilent, 2mL, Cat # 5182-0543 or equivalent)
- Amber (Agilent, 2mL, Cat # 5181-3376 or equivalent)
- Clear (Agilent, 0.8mL, Cat# or equivalent)

Kimwipes
Pasteur pipette
Porcelain spot plate

Scissors
Spatula
Teflon crimp (top) caps
Silver (Agilent, Cat # 5181-1210 or equivalent)
Blue (Agilent, Cat # 5181-1215 or equivalent)
Various Class A glassware
Volumetric flask (range 10mL to 50mL)
Weighing dish (VWR, Anti-Static, Cat # 89106 or equivalent)
Weighing paper (VWR, Cat # 12578 or equivalent)

Equipment

Analytical Balance (range 0.0001g to 1.0g)
GC with FID (Agilent, Model # 7890 Series or equivalent)
GC/MS (Agilent, Model # 5975 Series or equivalent)
Ultraviolet (UV) Lamp

Reagents

95% Ethanol (
Chloroform (JT Baker, ACS Grade, Cat # 9180 or equivalent)
Deionized water (in-house)
Hydrochloric acid (JT Baker, ACS Grade, Cat # 9535 or equivalent)
Methanol (JT Baker, ACS Grade, Cat # 9070 or equivalent)
p-dimethylaminobenzaldehyde (

Standards

Cocaine hydrochloride (USP, Cat # 14300 or equivalent)
Codeine phosphate (Grace-Alltech, Cat # 01801 or equivalent)
Lysergic acid diethylamide (Grace-Alltech, Cat # 01809 or equivalent)
Lysergic acid methylpropylamide (Grace-Alltech, Cat # 01810 or equivalent)

8. Safety

Due to the potential hazards, appropriate precautions should be taken as necessary. This includes, but is not limited to, the use of fume hoods, gloves, masks and safety glasses. Lab coats are to be worn at all times in the unit, unless performing administrative duties.

9. Reagent Preparation

Ehrlich's Reagent
Dissolve 2.5g of p-dimethylaminobenzaldehyde and bring to volume with 50mL of 95% Ethanol. Mix the solution until completely dissolved. Solution must be protected from light.

Lysergic Acid Diethylamide (LSD) Standard
Dissolve 2.0mg of lysergic acid diethylamide and bring to volume with 1.8mL of methanol. Mix the solution until completely dissolved.

Lysergic acid methylpropylamide (LAMPA) Standard
Dissolve 2.0mg of lysergic acid methyl propyl amide and bring to volume with 1.8mL of methanol. Mix the solution until completely dissolved.

Cocaine/Codeine Standard (QC Mix)
Dissolve 10.0 mg of cocaine hydrochloride and 10.0mg of codeine phosphate and bring to volume with 10mL of methanol. Mix the solution until completely dissolved.

10. Procedure

A. Evidence Handling

i. Evidence Officer will randomly assign sample to a chemist.
ii. The chemist will perform an evidentiary check on the sample. They will verify that the manila envelop, control card and the evidence correspond. They will observe the integrity evidence bag and its contents.
iii. Once the sample has being verified, the chemist will take custody of the samples by signing out the evidence in the chain of custody logbook.
iv. The sample will be brought to the chemist work area and stored in a secure manner at all times.
v. Upon analysis of each sample, the chemist will document all observations on the Drug Analysis Form.
vi. The information on the Drug Analysis Form will contain but not limited to the sample number, submitting agency, verification of the evidence gross weight, number of samples, container, description of sample, gross, package and net weight, ballistics notation, chemist notations and results, preliminary and confirmatory findings.

B. Sampling Plan

i. (to be determine)

C. Sample Preparation

i. For powders and liquids, no preparation is needed.
ii. For tablets (microdots), crush ½ of the tablet into a powdered form.
iii. For sugar cubes, crush and grind ¼ of the sugar cube into a powdered form.
iv. For paper squares, cut paper squares into tiny (1mm) pieces.

D. Color Test

i. For powders, tablets and sugar cubes, place 1-2mg of the sample into a labeled culture tube. For liquids, place 1-2 drops of the sample into a labeled culture tube. For paper squares, place 10-20 pieces of the sample into a culture tube.
ii. Add 1-3 drops of ehrlich's reagent to the sample and agitate the culture tube.
iii. Add 1-3 drops of concentrated hydrochloric acid to the sample and agitate the culture tube.
iv. The results will be recorded on the Drug Analysis Form by documenting the actual color/s observed. Negative observations will be recorded by stating no reaction or no color change

E. Interpretation

i. Ehrlich's reagent: Formation of a blue/purple color indicates the possible presence of LSD.

F. Ultraviolet Fluorescence Test

i. For powders, tablets and sugar cubes, place 1-2mg of the sample into a labeled culture tube. For liquids, place 1-2 drops of the sample into a labeled culture tube. For paper squares, place 10-20 pieces of the sample into a labeled culture tube.

ii. Place 1ml of methanol into the culture tube with the sample.
iii. Vortex the culture tube and let stand for 1 day. Cover the culture tube with parafilm.
iv. Remove 1-2 drops of the methanolic extract onto a filter paper and allow to dry.
v. Place the remaining methanolic extract into a labeled GC vial and cap tightly.
vi. Observe the filter paper under long wavelength UV light (360 nm).
vii. The results will be recorded on the Drug Analysis Form by documenting the actual color/s observed. Negative observations will be recorded by stating no reaction.

G. Interpretation

i. UV Test: Fluoresce of a blue/purple color indicates the possible presence of LSD.

H. Gas Chromatography Screen (as necessary)

i. The methanolic extract from section (F) can be used for the GC analysis.
ii. Initiate auto sampler sequence using the GENSCAN method running a blank solvent between each unknown sample and reference standard/s.
iii. Compare retention time of the each sample with the reference standard/s. Also check the chromatograph to determine if the sample needs to be diluted or concentrated.
iv. Positive GC analysis will be recorded on the Drug Analysis Form by the use of a plus (+). The result is considered positive when the retention time of the sample and the reference standard meet the laboratory criteria and are specified in the notes. Negative observations will be recorded by the use of a negative (-).

I. Gas Chromatography/Mass Spectrometry

i. Perform a preventative maintenance on the instrument by cleaning the injection port and changing the liner.
ii. Confirmatory analysis can be performed using the GC vial from the previous section (H).
iii. Initiate auto sampler sequence using the LSD method running a blank solvent between each unknown sample and reference standard/s.
iv. Compare retention time and ion spectra of the each sample with the reference standard/s (LSD and LAMPA).
v. Document the date analyzed and results of the GC/MS onto the MS Tracking Sheet, Drug Analysis Form and Control Card.

J. Criteria for Gas Chromatography/Mass Spectrometry

i. Retention time of the sample must be within +/- 1.5% of the reference standard.
ii. Library spectra match must be > 90%.
iii. There must be a visual spectral match between the reference standard and the sample.
iv. At least 5 of the major ions must be present for the sample.

11. Documentation

A. All results will be documented on the Drug Analysis Form.
B. All raw data will be generated and filed according to the laboratory policy.
C. A certificate of analysis will be generated for each lab number which will document the results.

12. Attachments

GC Method

GC/MS Method